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FLAMMABILITY ASSESSMENT TESTS FOR ORGANIC MATERIALS - THE OXYGE--ETC(U)
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FLAMMABILITY ASSESSMENT TESTS FOR ORGANIC MATERIALS - THE OXYGEN INDEX/TEMPERATURE INDEX CONCEPT

DOMENIC P. MACAIONE and RICHARD DOWLING, II
POLYMER AND CHEMISTRY DIVISION

September 1977

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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ABSTRACT

A series of organic polymers, composites, and polymeric foams have been investigated in terms of the Oxygen Index Method (ASTM-2863-74). The change in oxygen index as a function of temperature was determined for each material between ambient and 300 C and data plotted to prepare a temperature index profile.

CONTENTS

INTRODUCTION

Organic materials of present or potential military significance, by the very nature of their end use in military hardware, must be capable of resistance to a flammability threat of a greater or lesser degree. These materials should be selected to minimize hazards to personnel and optimize equipment survivability. It is, therefore, of interest and benefit to the Army to employ a flammability test method which is reliable and relatively uncomplicated to evaluate the flammability characteristics of polymeric materials.

Until recently, the only rapid, uncomplicated methods of evaluating the flammability of polymers were the so-called "bunsen burner tests" which were not always reliable. However, the oxygen index flammability test introduced by Fenimore and Martin[1] in 1966, and the extension of this method to materials at elevated temperature as reported by Routley[2] in 1973, has increased the reliability factor without undue complication.

The method of Fenimore and Martin defines the oxygen index n of a material as the percentage concentration of oxygen in an oxygen-nitrogen mixture which will just sustain equilibrium burning conditions, i.e., the heat produced by combustion of the sample balances the heat loss to the surroundings. It is the lowest concentration of oxygen which will support combustion, and is calculated from the equation:

$$n = O_2/O_2 + N_2$$

where n = oxygen index, O_2 = oxygen concentration, N_2 = *nitrogen concentration.*

Routley employed the oxygen index method to investigate the flammability behavior of materials and concluded that the method was of value in discriminating between materials in terms of flammability behavior. However, he discovered anomalous behavior in working with materials having an oxygen index above 20.8 (the oxygen concentration of air) which would not be expected to burn in normal atmosphere. Routley found that materials with an oxygen index of 27 or 28 would often burn in air and he attributed this behavior to thermal or geometric considerations.

In an effort to evaluate flammability behavior at temperatures above ambient, Routley determined the oxygen index in a heated oxygen-nitrogen atmosphere and found that he could ignite and burn most materials at progressively lower oxygen concentrations as the temperature of the surrounding atmosphere increased. He defined "temperature index" as that temperature where the curve of a material's temperature dependence of oxygen index intercepts an oxygen concentration of 20.8. This is also referred to as Tox-21. Thus, the utility of the oxygen index test was extended in terms of evaluating the flammability behavior of materials, but at the same time the reliability of the test was brought into question.

It would seem that in the interest of evolving a test method which produces a numerical value, or rating, of flammability the use of the temperature dependence

1. FENIMORE, C. P., and MARTIN, F. J. *Candle-Type Test for Flammability of Polymers.* Modern Plastics, v. 44, no.3, November 1966, p. 141.
2. ROUTLEY, A. F. *Development of the Oxygen Index Concept for the Assessment of the Flammability Characteristics of Materials.* Central Dockyard Laboratory, H. M. Dockyard, Portsmouth, England, CDC 5/73, November 1973.

curve has been overlooked. If this curve is considered a profile of material flammability behavior it could yield useful information by means of which the flammability response of organic polymers could be evaluated.

Investigation has shown that although two materials have similar oxygen index values at 25 C, the shape, or profile, of the temperature dependence curve may be quite different for each type of material. Thus, a polymer with acceptable flammability characteristics at ambient temperature could become a hazard at an elevated temperature as, for example, in the presence of a fire situation, although it may not be the initial source of the fire.

With these factors in mind the objective of the project reported herein has been to evaluate the oxygen index and temperature index methods to determine their utility for the assessment of flammability behavior of polymeric materials of interest to the Army. Answers to the following questions were sought:

a. In routine operation can the oxygen index test truly differentiate between polymer classes?

b. Can the oxygen index test be applied by the Army to specifications for procurement of polymeric materials?

c. Does the temperature index test afford a more reliable picture of flammability behavior and, if so, could it be employed to better advantage than the oxygen index test in military specifications?

EXPERIMENTAL

Instrumentation

The first phase of the program was directed toward either the fabrication of the required apparatus or the acquisition of a commercial unit which would accomplish the desired testing. A commercial instrument (Michigan Chemical Company, Oxygen Index Smoke Densitometer, Model 1300) was obtained. The instrument is schematically described in Figure 1. In essence, the apparatus consists of gas flow meters for the introduction of oxygen and nitrogen, in known amount and at a controlled flow rate, into a pyrex column. The premixed gases are passed through a bed of inert material to insure complete mixing and uniformity of the gaseous atmosphere.

In the elevated temperature mode required for temperature indexing, the gas stream is warmed by an electrically heated coil at the base of the column. The temperature level and rate of heating are regulated by a voltage controller and the temperature is monitored by an external digital temperature indicator (Doric, Model 412, Temperature Trendicator) employing an iron-constantan thermocouple, capable of ±0.1 C resolution. The apparatus was modified to permit the introduction of laboratory air from a compressed air line which was reduced to 20 psi delivery pressure to conserve oxygen and nitrogen supplies while protecting the heating coil with a constant flow of gas during periods of heating and cooling.

Test Procedure

Oxygen index values for the various samples examined were determined at ambient (25 C) temperature according to the criteria set forth in ASTM-D 2863-74 "Flammability of Plastics Using the Oxygen Index Method". Temperature index values were determined by applying the same criteria to samples placed in an oxygen-nitrogen environment at temperatures of 100, 200, and 300 C. The values of oxygen index thus obtained over the range from ambient to 300 C were graphed versus temperature to obtain the temperature dependence of oxygen index or the so-called "temperature index profile".

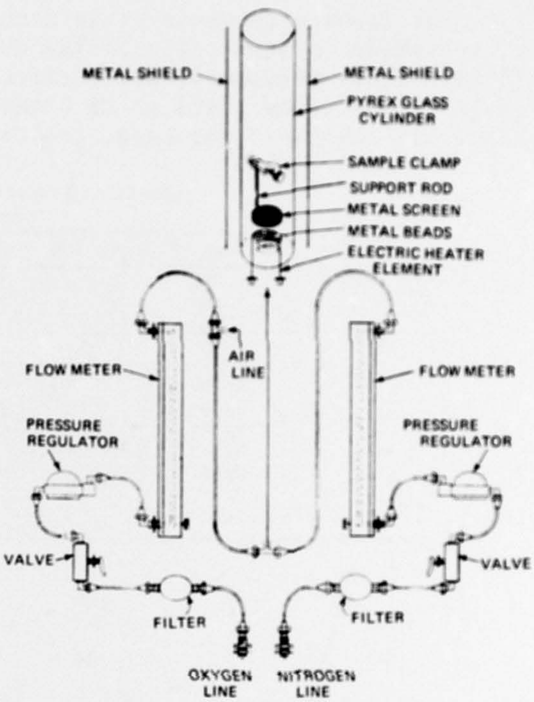

Figure 1. Schematic of apparatus.

RESULTS AND DISCUSSION

Polymer specimens selected for testing were of four types:

a. unfilled solid polymers;
b. solid polymers filled with short glass or carbon fibers;
c. composite materials composed of a resin matrix and fiber reinforcement; and
d. polymeric foams.

The samples were selected to represent general classes of polymeric materials having present or potential interest to the Army in end-item applications.

Initial oxygen index determinations were made with poly(methylmethacrylate) having an oxygen index of 17.3 ±0.2, which has been accepted as a secondary standard to insure that the apparatus was operating properly. This experiment was followed by determination of the oxygen index of a polysulfone sample at 25 to 300 C. Based upon 40 separate determinations it was concluded that the oxygen index value could be determined within ±1.0% oxygen. A similar experiment was carried out with an epoxy/glass composite material since the presence of a filler material can result in erratic burning of the sample, and once again the oxygen index value could be determined within 1.0% oxygen.

The results of these experiments indicate that with proper attention to detail, materials can be evaluated with a reasonable degree of precision. However, it should be remembered that such factors as individual sample uniformity, composition, char formation, evolution of gaseous components, etc., may alter the behavior of a sample under examination, and the accuracy of the results. (The occurrence of molten dripping during the test is a specific example of behavior which can give extremely misleading values of oxygen index.)

Unfilled Polymers

The results shown in Table 1 and Figure 2 represent the behavior of a series of polyamide (nylon) resins. The chemical formulae of the repeat units are shown to illustrate structural similarity. Except in the case of Nylon 12, the simple oxygen index value (data at 25 C only) indicates little difference in flammability behavior. On the other hand, the data obtained by temperature indexing from 25 to

Table 1. TEMPERATURE DEPENDENCE OF OXYGEN INDEX FOR POLYAMIDE RESINS

Temp °C	Nylon 6/6	Nylon 6/10	Nylon 6	Nylon 11	Nylon 12
25	31.2	32.6	31.8	32.8	36.2
100	30.0	30.0	28.9	28.8	29.5
200	20.3	27.8	26.2	24.1	28.3
300	17.9	22.4	20.2	17.5	20.3

Nylon 6/6 $[-NH-CH_2)_6-NH-CO(CH_2)_4CO-]_n$
Nylon 6/10 $[-NH-(CH_2)_6-NH-CO(CH_2)_8CO-]_n$
Nylon 6 $[-NH(CH_2)_5-CO-]_n$
Nylon 11 $[-NH(CH_2)_{10}CO-]_n$
Nylon 12 $[-NH(CH_2)_{11}CO-]_n$

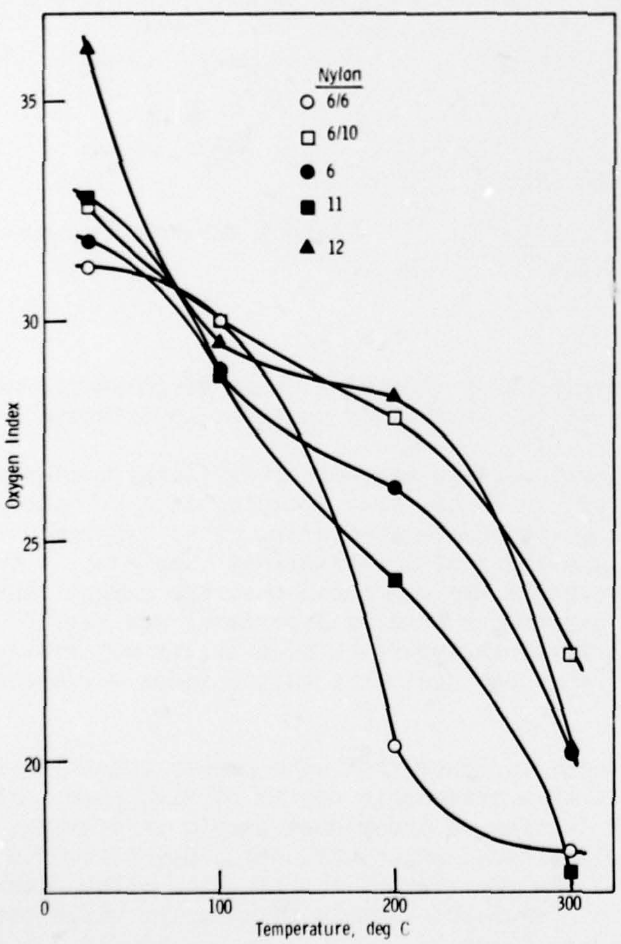

Figure 2. Temperature dependence of oxygen index.

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300 C indicates that each of these materials responds in a different manner as the thermal environment is changed. Although Nylon 12 has the most acceptable value at 25 C, it is no longer the best selection at 300 C, nor is Nylon 6/6, with the lowest value at ambient temperature, the least acceptable at 300 C.

Table 2 and Figure 3 present the results obtained from polymers of different molecular structure and illustrate the effect of temperature dependence of oxygen index versus chemical composition. These polymers possess similar oxygen index values at 25 C but they do not behave similarly in their response between 25 and 300 C. The results of temperature indexing shows that polypropylene and Nylon 11 have similar responses, that the phenolic resin has several "crossover" points, and that the bis-(phenoxy)phosphazene shows the least tendency toward ignition and burning from ambient to 200 C with quite acceptable behavior from 200 to 300 C. The data thus obtained on unfilled organic polymer specimens indicates that the temperature index profile is a more informative measure of flammability behavior than the single oxygen index determination at ambient temperature.

Table 2. TEMPERATURE DEPENDENCE OF OXYGEN INDEX EFFECT OF CHEMICAL STRUCTURE

Temp °C	Isotactic P.P.	Nylon 11	Phenolic Resin	Poly[bis-(phenoxy)-phosphazene]
25	31.8	32.8	35.7	32.0
100	27.8	28.8	28.0	30.5
200	20.3	24.1	25.7	26.3
300	17.1	17.5	24.5	23.9

Filled Polymers

The results presented in Table 3 and Figure 4 consider the effect of filler on the flammability of polymeric materials. Two polymers were filled with carbon fiber at equal levels, but they responded differently to a combustion situation. The bisphenol-A polysulfone tends to be somewhat more flame resistant with increased loading, while polyphenylene sulfide remains essentially unchanged with up to 30% loading. Two other polymers, a polyurethane and a styrene-acrylonitrile copolymer, were glass filled at the 40% level. The filler

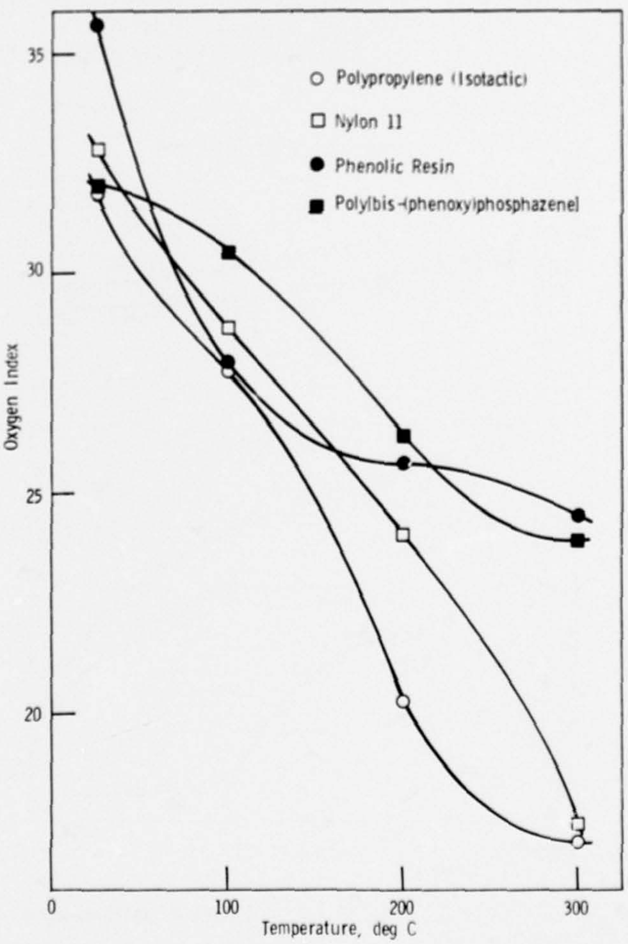

Figure 3. Temperature dependence of oxygen index versus chemical structure.

alters the flammability character of each polymer but in the case of the copolymer it does so in a manner which would not have been predicted from the data at ambient temperature.

These results further illustrate that the temperature index profile gives a more complete and at times unexpected insight into flammability of organic materials.

Composite Materials

Data obtained by application of the temperature indexing concept to composite materials consisting of epoxy resin matrices and various fibers is shown in Table 4 and Figure 5. Composites a and d are commercial materials while composites b and c

Table 3. TEMPERATURE DEPENDENCE VERSUS LOADING

Temp	(a)			(b)			(c)		(d)	
(deg C)	0%	20%	30%	0%	20%	30%	0%	40%	0%	40%
25	29.6	33.4	35.5	42.9	41.1	41.6	22.2	20.4	18.2	19.3
100	30.7	31.9	34.6	41.7	40.3	40.7	20.7	18.2	17.0	18.2
200	27.0	31.9	33.3	41.0	39.8	40.1	19.0	18.2	17.0	16.6
300	26.7	29.4	30.0	40.5	39.7	40.0	18.6	15.8	16.2	16.2

(a) Bisphenol A polysulfone - Carbon fiber
(b) Polyphenylene sulfide - Carbon fiber
(c) Polyurethane - Glass fiber
(d) Styrene acrylonitrile - Glass fiber

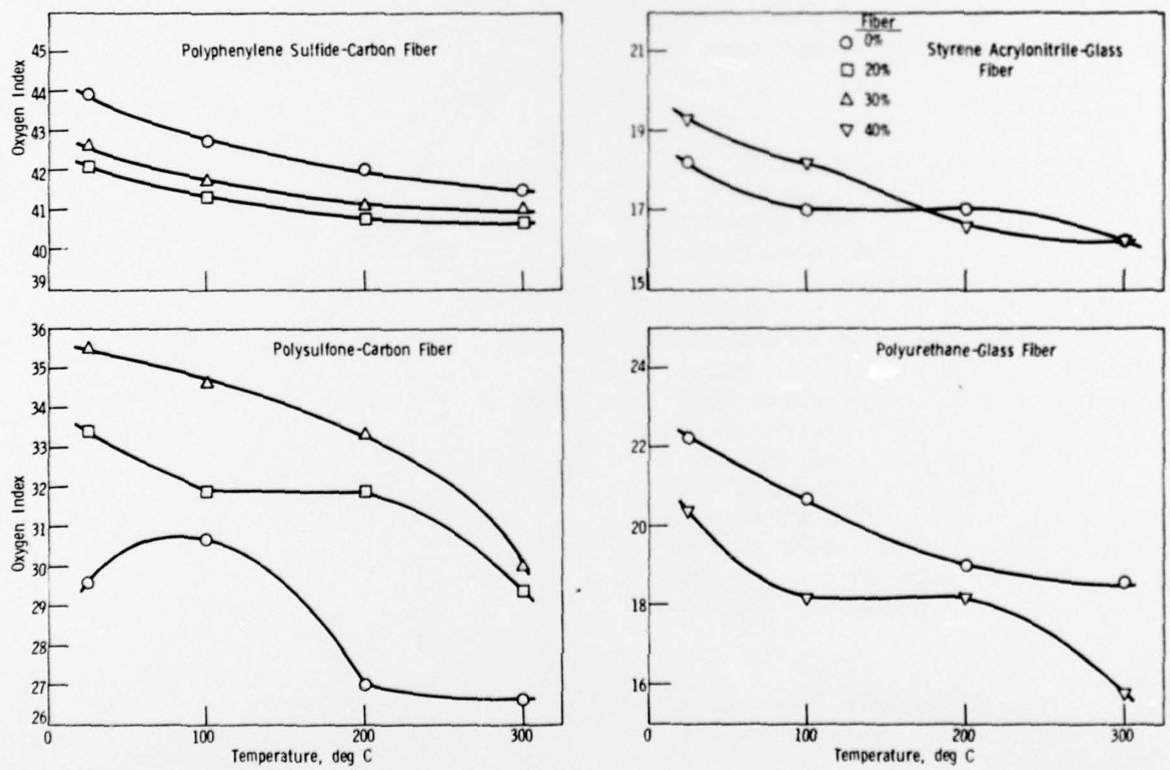

Figure 4. Temperature dependence versus loading.

were prepared in-house from commercial components. In this experiment it is possible to detect Tox-21 which Routley refers to as the material's temperature index, thus indicating the temperature at which the sample would combust in air. Although the temperature index profile affords an overall indication of the behavior of an organic material at elevated temperature, the Tox-21 value could be used to "merit rate" materials at temperatures above ambient. The problem at this time is that within the use limits of presently available apparatus many polymers, especially those in the high performance class, do not drop sufficiently to detect a value of Tox-21. In this respect, as well as the earlier comments concerning temperature indexing, the temperature dependence profile is a more informative measure of materials response above ambient temperature.

Polymeric Foams

The data presented in Table 5 and Figure 6 are the results of temperture index experiments with organic materials used in structural foam applications. The urethane and methacrylimide foams are in commercial use while the poly(benzimidazole) and carbon fiber-reinforced polyimide foams are of an experimental or developmental nature, being intended for high performance applications. As the use of foam materials increases, data such as this will become vital in assessing high temperature performance in response to a flammability threat.

Table 4. TEMPERATURE DEPENDENCE OF COMPOSITE MATERIALS

	Epoxy Novolac		Epoxy Resin	
Temp (deg C)	Glass Fiber (a)	Kynol* Fiber (b)	Kevlar 49† (c)	Carbon Fiber (d)
25	41.2	22.0	26.4	24.7
100	39.2	20.8	25.3	23.2
200	26.2	18.2	24.4	18.5
300	23.1	17.3	19.1	18.0

*Trademark American Kynol, Inc., Niagara Falls, New York 14302
†Trademark E. I. DuPont, Inc., Wilmington, Delaware 19898

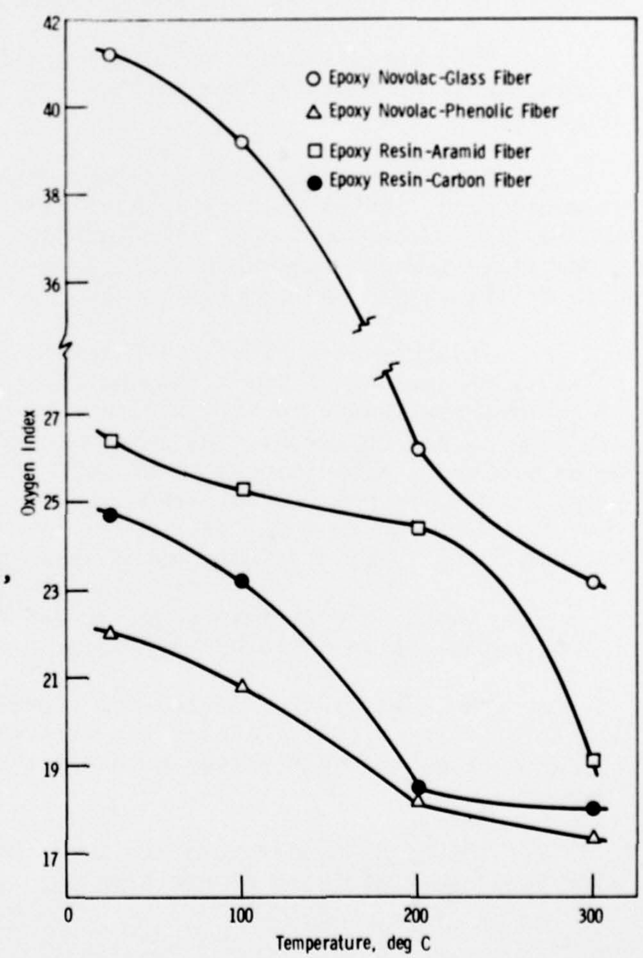

Figure 5. Temperature dependence of composite materials.

Table 5. TEMPERATURE DEPENDENCE OF POLYMERIC FOAM MATERIALS

	Oxygen Index			
	(a)	(b)	(c)	(d)
Temp (deg C)	Urethane	Methacryl-imide	Poly(benz-imidazole)	Carbon Fiber-Reinforced Polyimide
25	22.5	22.0	63.6	59.5
100	21.0	17.9	62.0	54.7
200	18.0	16.2	61.7	54.5
300	16.4	14.0	61.7	51.9

CONCLUSIONS

At the initiation of this project it was intended that the oxygen index test and its extension to include the temperature index concept be evaluated in terms of the questions set forth in the Introduction of this report. Having applied the test procedure to several materials which are representative of those of interest to the Army, our results indicate the following.

a. The oxygen index test can generally be relied upon to give a first-order approximation of polymer flammability. It cannot, by itself, differentiate classes of polymers.

b. Because the oxygen index test is essentially a single point measurement it would have limited utility as a military specification parameter, and should not be applied as such. Our data indicate that the values obtained at ambient temperature are not generally indicative of flammability behavior as the temperature of the sample environment is changed.

c. Incorporation of the temperature index procedure does present a more reliable picture of polymer flammability behavior since it considers the response of the material under test to an elevated temperature environment. Data points can be obtained at any desired interval and in most cases a value for Tox-21 can be determined. With respect to military specifications it would seem that a Tox-21 value should be an included parameter. As more data become available one may find that the temperature index profile is characteristic of the polymer class; however, further investigation on this point is required.

Over the course of this study it has become evident that certain changes in apparatus design would be advisable.

a. The heat control system for elevated temperature work would be more reliable if a proportional controller were used to cycle only a small percentage of the thermal load and better maintain the working temperature in the sample chamber.

b. The upper temperature limit of the apparatus should be extended to permit more complete evaluation of new high performance materials.

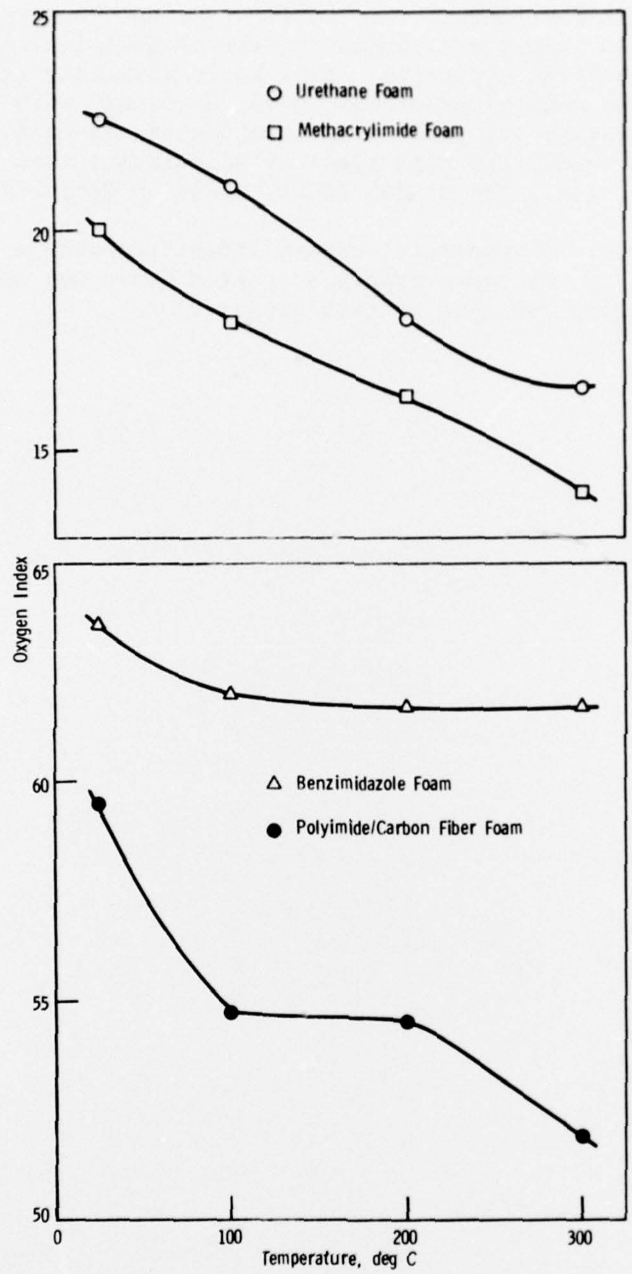

Figure 6. Temperature dependence of polymeric foam materials.

c. A reliable instrumental method for sensing the oxygen concentration in the oxygen-nitrogen atmosphere, replacing the present flow meter system, should be incorporated into the apparatus. This would eliminate the necessity for the usual flow rate/gas volume conversion curves necessary with flow meters, and would eliminate the necessity for gauge recalibration at six-month intervals. Such a system would also reduce the time spent in calculating test results since the oxygen concentration, i.e., the oxygen index, would be determined directly.

(Author's Note: A commercial oxygen index/temperature index apparatus which incorporates most of the improvements suggested above has become available in the United Kingdom during the term of this project.)

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